

ORIGINAL ARTICLE

Influence of water sorption of the underlying abutment on fracture resistance of zirconia copings

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Abstract

Objective. To investigate the influences of abutment water sorption and various aging parameters on the fracture resistance of zirconia copings. **Methods.** Using a master die, identical replicas were made from three resin materials. The first was a melamine resin with very high water sorption ($n = 48$), the second an experimental resin core build-up composite with moderate water sorption ($n = 40$) and the third a commercially available core build-up composite with low water sorption ($n = 40$). On the abutment replicas, zirconia copings ($n = 128$) were made using a computer-aided design–computer-aided manufacturing system. The copings were luted onto the abutments using zinc oxide phosphate cement. In the melamine group, a subgroup of samples ($n = 8$) was cemented with a composite cement as controls. The forty specimens in every abutment material group were randomly divided into one of five subgroups, as follows: (i) not aged; (ii) mechanically (dry) loaded only (50 N; 1.2×10^6 cycles); (iii) stored for 10 days in water; (iv) thermally cycled (TC; $6000 \times 5/55^\circ\text{C}$); and (v) TC and mechanically loaded (TCML; 50 N, 1.2×10^6 ; $6000 \times 5/55^\circ\text{C}$). After aging, all copings were loaded to fracture. **Results.** A statistically significant difference was found between the three abutment–die groups if the samples were aged by TCML. The zirconia copings cemented on abutments with high water sorption fractured during TCML, and the subgroup with moderate water sorption had significantly lower fracture resistance. A change of luting material had no impact on this behavior. **Conclusion.** Only the simultaneous combination of all chosen aging factors (TCML) was able to detect a difference in fracture behavior of a zirconia coping luted on abutments with varying water sorption.

Key Words: *Abutment, core build-up, mechanical loading, resin, thermal cycling, water sorption, zirconia*

Introduction

Hygroscopic expansion of resin-based core build-up materials or luting agents has an impact on the fracture resistance of glass–ceramic restorations. For example, Sindel et al. [1] demonstrated that hygroscopic expansion could propagate cracks in glass–ceramic restorations if they were immersed in water. The water sorption of various dental materials is a process which depends on the composition of the material and the duration of water exposure.

Literature reports have described conflicting results about the time needed to achieve an equilibrium state of water sorption in a dental resin composite. Some studies showed that this state was reached after 7 [2] or 14 days [3], while others found that water uptake could continue for as long as 3 years [4]. Water

sorption of composites is controlled by diffusion and depends mainly on the composition of the resin matrix. Hydrophilic constituents such as hydroxyethyl–methacrylate (HEMA) or other moieties which contain hydrophilic groups increase water uptake. The amount of water sorption furthermore depends on the filler content, the filler type [5] and the size of the specimens, which have been evaluated [6]. All these factors may explain the conflicting results which have been presented regarding the water sorption of resin-based materials.

Independent from the discussion about the amount of, duration of or reason for water uptake of resin-based materials, all ceramics can fail if the underlying structures (core build-up or luting agent) show hygroscopic expansion. Zirconia has better mechanical properties (e.g. strength) than glass ceramics. It is

therefore to be expected that zirconia will not be affected by the hygroscopic expansion of core build-up or luting agents.

This study investigates how underlying abutment materials with various hygroscopic expansions influence the fracture resistance of zirconia copings. Furthermore, the impact of single aging factors such as (dry) mechanical loading only, water storage and thermal cycling on the fracture resistance of zirconia copings was evaluated, together with the combination of these factors (i.e. mechanical loading and thermal cycling procedures). The hypothesis was that the water sorption of core build-up would have no impact on the fracture behavior of zirconia copings, while the type of aging procedure would have an impact.

Material and methods

Construction of identically shaped artificial abutment teeth

A master die was prepared using an artificial melamine resin tooth 36 (Frasaco Company, Tettang, Germany). Melamine resin is a hard, thermosetting resin material made by the polymerization of melamine and formaldehyde. It is an organic resin based on cyanamide, with a 1,3,5-triazine skeleton. Melamine teeth are produced using an injection molding process.

The tooth preparation followed the recommendations for zirconia all-ceramic crowns [7]. A deep chamfer finish line was prepared (0.8 mm) and a circular and occlusal anatomical reduction of 1.5 mm was carried out with a preparation angle of 6°. Sharp edges and undercuts were eliminated.

The master die was copied using a Celay™ copy-milling machine (Mikrona, Spreitenbach, CH) [8]. While a small steel ball scanned the entire surface of the master die, a bar linkage led the drills of the milling device and formed a copy of the master die. Three materials with different degrees of water sorption were chosen to construct the master die copies. The first material was a melamine resin, which had the largest amount of water sorption. Although melamine resins are not clinically used in dentistry (because of their water sorption) this material was chosen to demonstrate the possible effects of hygroscopic expansion on zirconia copings [9,10]. This group was considered as a positive control group (4 × 8 dies of Frasco were made). The second group was milled from the core build-up material Rebuilda™ (Voco, Cuxhaven, Germany). Rebuilda is a high filled bisphenol-glycidyl-methacrylate-based composite (>60 Vol %) with low water sorption (3 × 8 dies of Rebuilda were made). The third group was based on an altered Rebuilda material which had 30% greater water sorption. This experimental material was mixed by Voco. In the following, this experimental material is named Rebuilda-30 (3 × 8 dies of Rebuilda-30 were made).

Zirconia copings

We constructed 128 zirconia copings using the Cercon™ (DeguDent, Hanau, Germany) computer-aided design–computer-aided manufacturing (CAD-CAM) system. Scan powder was thinly sprayed on the abutments and then every abutment was scanned using the Cercon Eye™. The scan was sent to a computer and a coping was constructed on the computer screen. The data that were sent to the Cercon Brain™ milling machine consisted of parameters such as a coping wall thickness of 0.6 mm, a cement layer thickness of 20 μm and a spacer area of 80%. Using a pre-sintered yttrium-stabilized zirconia blank, the milling machine prepared a coping that was ≈30% bigger than the final coping. The copings were finally sintered in a Cercon Heat™ oven at a temperature of 1350°C for 7 h. The fit of the copings was checked using the polysiloxane Fit-checker™ (Voco). If necessary, minor corrections were made with diamond burs under water cooling. Copings that needed major adjustment were replaced by new ones.

The copings were randomly assigned to subgroups each containing eight copings. As a control, one group was luted onto Frasco dies using the composite cement Variolink II (Ivoclar-Vivadent, Schaan, Lichtenstein). All other groups were luted with the zinc oxide phosphate cement Harvard™ (Harvard, Berlin, Germany). All in all we investigated three different groups of abutment materials and these three groups were further assigned to subgroups which underwent five different aging procedures (Figure 1).

Aging procedures

The following aging procedures were performed (Figure 1) [11,12]:

- (1) No aging (control).
- (2) Storage for 10 days in distilled water at 37°C (WS).
- (3) Thermal cycling: 6000 cycles at 5°/55°C, changed every 2 min, for 8.3 days (TC).
- (4) Mechanical loading: 50 N; 1.2×10^6 cycles at 1.6 Hz for 8.3 days.
- (5) Thermal and mechanical loading: 50 N; 1.2×10^6 cycles at 1.6 Hz for 8.3 days; 6000 cycles at 5°/55°C, changed every 2 min, for 8.3 days (TCML).

After aging, the samples were loaded to fracture using the universal testing machine Zwick 1446™ (Zwick, Ulm, Germany). A 0.3-mm thick tin foil was placed onto the occlusal surface of the cemented copings. The load to fracture was applied using a steel ball of 12-mm diameter. The load force during cracking (10% reduction in actual load force) was recorded [11].

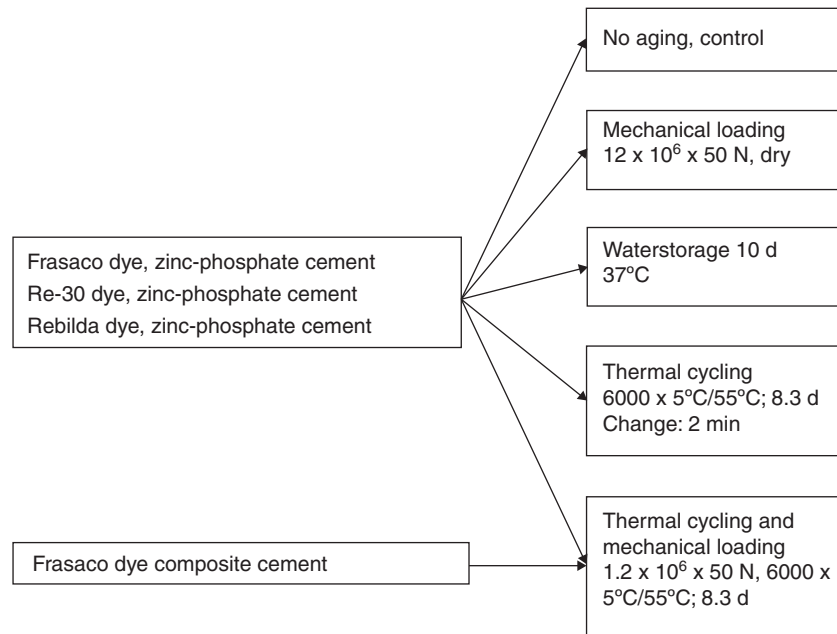


Figure 1. Overview of the composition of the test groups.

Water sorption

The weight of eight specimens of each type of abutment die was measured using a Sartorius laboratory beam balance type 160 PD1™ (Sartorius, Göttingen, Germany). The dry specimens were weighed at room temperature. A second measurement was done after storing the samples in distilled water for 10 days at 37°C in an incubation box. The weight difference as a result of water storage was determined [13].

Analysis of fracture fragments

The fracture fragments of the coping were analyzed using a scanning electron microscope (Quanta FEG 400; Fei Company, Eindhoven, the Netherlands). The aims were (i) to determine whether the origin of fracture started on the ‘cement side’ or the ‘occlusal side’ of the copings; and (ii) to evaluate the quality of the manufacturing process of the copings. Copings that failed due to manufacturing mistakes were detected and eliminated from further investigations.

Overview pictures were made under a low-pressure vacuum (0.08 Torr) at a magnification of $\times 25$ – 30 . Details of the fracture mirror were analyzed under a low-pressure vacuum at higher magnification (up to $\times 200$). The fracture mirror is considered to indicate the origin of the fracture. In ceramics, coarse microstructural elements or flaws may trigger early hackle lines within the mirror [14].

Statistics

Continuous variables are presented as means and standard deviations (SDs). The Mann–Whitney

U-test was used to evaluate statistical differences. The level of significance was set at $\alpha = 0.05$.

Results

Water sorption

The Frasco samples had $3\% \pm 0.6\%$ more weight after 10 days storage in water, compared to $0.8\% \pm 0.09\%$ more weight for Rebuilda-30 samples and $0.17\% \pm 0.05\%$ more for Rebuilda.

Fracture resistance of the copings

Without any aging procedure, copings showed no statistically significant difference in their mean values of fracture resistance (Figure 2, Table I). Their fracture resistances were independent of the type of underlying abutment material.

Independent of the core build-up material, we found comparable mean fracture resistances in the groups that underwent only thermal cycling or mechanical loading. However, the water storage group had a significantly higher fracture resistance for the Rebuilda-30 group ($P < 0.001$), while the Rebuilda and Frasco groups showed comparable fracture resistances. A statistically significant difference was found between the three abutment-die groups if the samples were aged by a combination of mechanical and thermal loading (TCML). Then, the copings in the Frasco group fractured during TCML. A change of luting material had no impact on this behavior. Specimens luted with the resin cement Variolink fractured during TCML, as did those luted using zinc oxide phosphate. The mean fracture

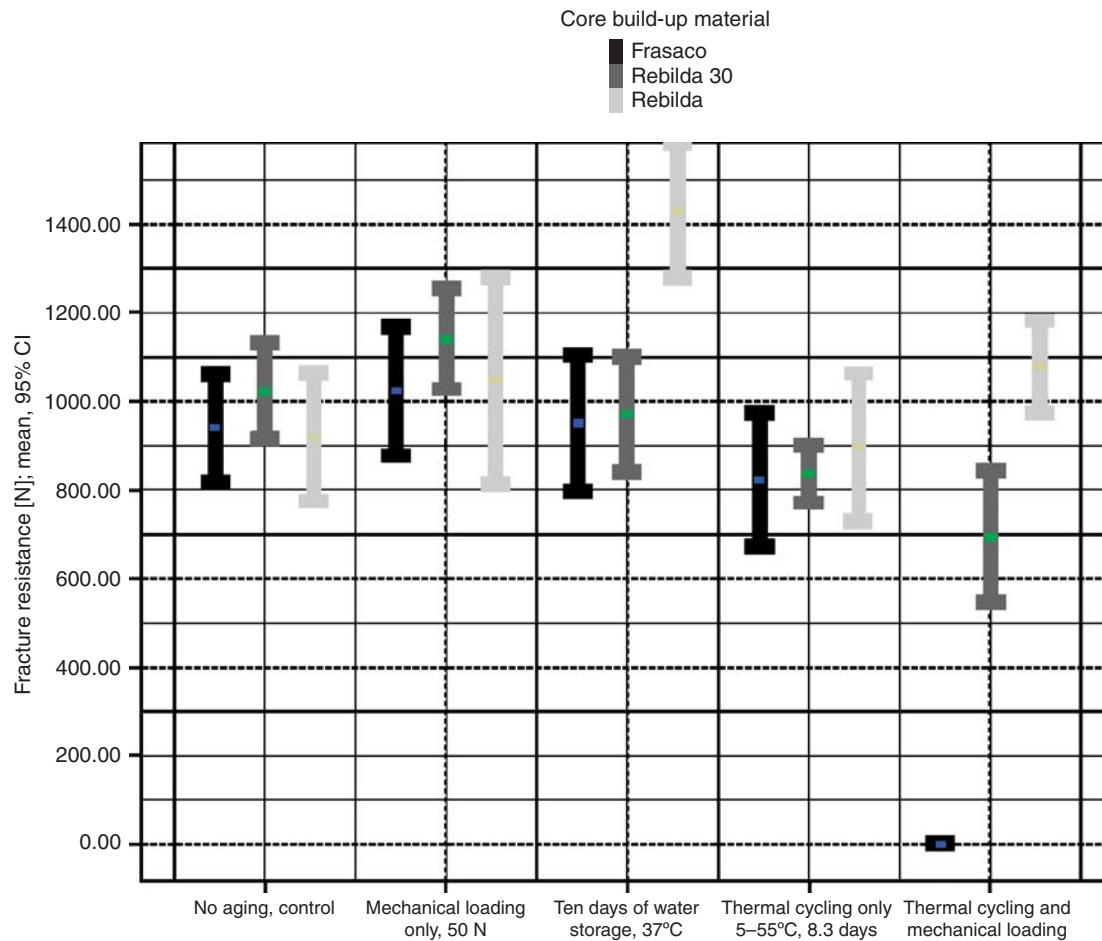


Figure 2. Means and 95% confidence intervals of the fracture resistance values.

resistance in the Rebuilda-30 group ($P = 0.036$) was found to be significantly lower than that in the Rebuilda group.

Fracture fragment analysis

Using scanning electron microscopy (SEM), analysis of the fracture fragments of the copings that failed during TCML revealed an homogeneous structure of the zirconia material. No hints were found that the failures during TCML were caused by manufacturing

mistakes, for example pollution of the mass or too low a sintering temperature (Figure 3).

The evaluation of the origin of fracture during the load-to-fracture test showed uniformly that the fracture started on the cement side. Fracture mirrors started from the cement side of the copings (Figure 4).

Discussion

The mechanical properties of zirconia greatly outmatch those of glass ceramic. Some authors have

Table I. Mean \pm SD of fracture resistance values (N) after different aging procedures of zirconia copings luted onto various abutment dies.

Aging procedure	Frasaco die	Rebuilda-30 die	Rebuilda LC die
Zinc oxide phosphate-luted			
No aging	941 \pm 146	1023 \pm 130	919 \pm 172
Mechanical loading	1023 \pm 174	1141 \pm 136	1046 \pm 279
Water storage	951 \pm 167	971 \pm 155	1090 \pm 189
Thermal cycling	823 \pm 179	835 \pm 77	896 \pm 198
Thermal cycling and mechanical loading	0 (failure during TCML)	695 \pm 178	1077 \pm 125
Composite cement-luted			
Thermal cycling and mechanical loading	0 (failure during TCML)		

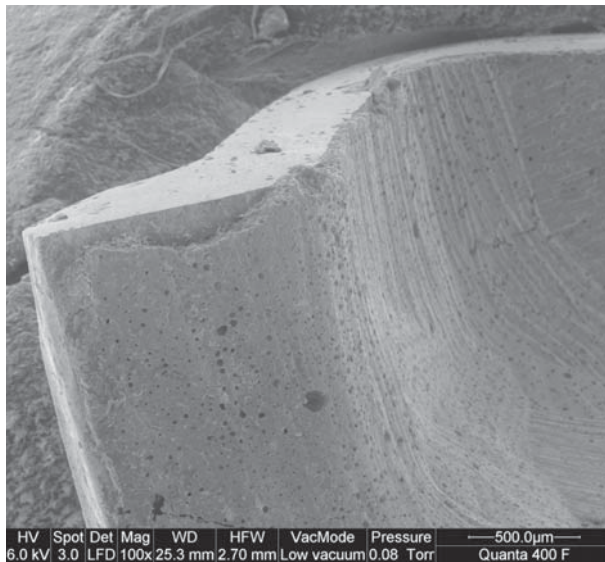


Figure 3. Fracture fragment of a zirconia coping. This coping fractured during thermal cycling and mechanical loading. The surface is covered with a thin layer of a zinc oxide phosphate luting agent.

described zirconia as ‘ceramic steel’ [15]. This term should indicate that zirconia is a material that performs well under any type of load. Therefore, it was surprising that the copings luted with zinc oxide phosphate cement onto the Frasco dies failed during TCML, because a lot of zirconia restorations had surpassed our masticator using identical loading parameters without significant failures [16].

Our first thought was that a mistake during the manufacturing procedure had caused the failure. However, the SEM analysis in Figure 3 demonstrated perfect sintered copings and showed no inclusion of any items which may have been responsible for early failure. The investigation of this group was repeated, but the results remained unchanged.

Our second idea was that the luting agent has an influence. However, a further identical group luted with the resin-composite cement (Variolink II) again showed coping failures during TCML.

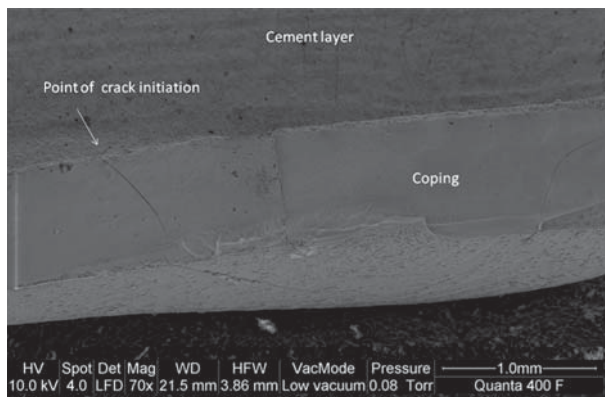


Figure 4. Fracture fragment of a zirconia coping after a load-to-fracture test.

Our next thought was that the water sorption of the melamine resin-type abutment dies may have been responsible for the observed failures. Melamine resin has high water sorption [9]. In this investigation, the melamine resin samples had about three times higher water sorption than that of Rebuilda-30 and 15 × times higher than that of Rebuilda. It has been reported that water sorption of core build-up materials can cause severe damage to glass–ceramic restorations [1]. However, to the best of our knowledge, this has not yet been described to be of importance for zirconia dental restorations. It is believed that zirconia (like alloys) is not affected by hygroscopic expansion of the core build-up or luting agent. In order to check this hypothesis, we separated out the factors that were applied to the specimens during TCML. In addition to a control without any aging, we investigated the influence of mechanical loading only, water storage, thermal cycling in water and the combination of thermal cycling and mechanical loading. The results demonstrated that individual factors had no impact if melamine resin abutment dies were used. However, if all the parameters were combined, the samples failed, independent of the type of luting agent used. When a further modification was made by using a core build-up material with lower hygroscopic expansion, the samples surpassed TCML without failures (Rebuilda-30 and Rebuilda groups). Furthermore, it could be shown for these groups that the lower the water sorption of the core build-up material, the higher the mean fracture resistances of the zirconia copings after TCML and loading to fracture. The conclusion to be derived from all these observations may be that hygroscopic expansion together with thermal cycling and mechanical loading can damage zirconia copings, but only in cases with high hygroscopic expansion. Normally, dental core build-up materials will not have such a high hygroscopic expansion as the zirconia copings that seemed to be affected. Even the experimental Rebuilda-30 material, which had considerably higher water sorption than ‘normal’ dental composites, showed no failures during TCML.

This investigation demonstrates that the term ‘ceramic steel’ [15] is misleading. Zirconia does not perform like a ductile metal alloy; it performs like a brittle material. Zirconia resists stress better than most glass–ceramic materials, but the dentist should still bear in mind that not everything is possible with zirconia, which does have some limitations. One aspect was not considered in this investigation. Normally, zirconia copings are veneered using feldspathic ceramic. The fracture behavior of two- or three-layer systems is more complex [17,18]. Therefore, the results cannot be transferred to veneered clinical zirconia restorations. However, the aim of this study was to investigate the behavior of zirconia itself using the chosen test design and not to evaluate complex

veneered crown systems. The behavior of zirconia itself will become of more interest in the future. The next generation of CAD-CAM systems will be able to mill not only zirconia copings but also complex occlusal structures. Initial investigations of complete zirconia crowns are promising. Then, the question of the amount of water sorption of abutments will become the focus of restorative dentistry.

Conclusions

In contrast to our expectation, even zirconia may be damaged by underlying abutment materials with high water sorption. However, this behavior of zirconia could only be detected if various thermal and mechanical parameters were combined in a simultaneous trial.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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